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CERAMIC SYSTEMS FOR MISSILE STRUCTURAL APPLICATIONS

31 July 1963

Prepared under Navy, Bureau of Naval Weapons

Contract NOw-63-0143-d

QUARTERLY REPORT NO. 3

1 May 1963 through 31 July 1963

Engineering Experiment Station
Georgia Institute of Technology
Atlanta, Georgia

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ABSTRACT

Techniques for flame glazing (surface melting) slip-cast fused silica were successfully developed using an oxy-acetylene flame. A high degree of parameter flexibility was obtained with this flame.

Requirements for producing strong dense ceramic-wood fiber felts using modified paper making techniques were determined on 1: 2: 2 wood fiber, alumina clay compositions. Highly dispersed and/or fibrillose fibers, a solid contents no greater than 2 w/o and a flocculating system were found necessary. Indications were that type of clay used affected drain time but had little effect on green density of felts. Strong alumina-clay compacts of 50 to 60 lb/ft^3 fired density were obtained. Linear shrinkage was 11 to 12 per cent, however, this should decrease with decreasing clay content.

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Quarterly Report No. 3, Project No. A-651

I. PURPOSE

The purpose of Contract No. NOw 63-0143-d is to perform a research and development program directed toward development of techniques to fully exploit the potential of readily available ceramic systems for use as structural components in hypersonic missile applications.

II. INTRODUCTION

A. Slip-Cast Fused Silica Radomes

To prevent an overrun of expenditures, the rate of effort under this contract has been reduced for the remainder of the contract year. This is in accordance to discussions with the Naval Technical Director of this contract. This reduction was necessitated by an earlier increase in effort expended in the preparation of radomes for rain erosion tests, but will not change the contract man-year effort.

The successful rain erosion test-results of slip-cast fused silica has provided sufficient information for groups within the Naval Bureau to evaluate this material and continue the work initiated under this contract.[1,2] Therefore, the slip-cast fused silica radome work has been abandoned for the remainder of the contract year. Emphasis will be placed on felted ceramic structures.

The contractor has recently received a contract from the Air Force, Contract No. AF 33(657)-11504, Design and Development of an E-M Window for Air Lift Reentry Vehicles, which, in part, will parallel the work under this Project. The data on slip-cast fused silica radomes obtained under this contract were to a large extent responsible for the Air Force's interest in supporting related work for their particular problem area.

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(1) Quarterly Report No. 2, Page 18.

(2) Hallse, R. L., "Rain Erosion Resistance of Slip-Cast Fused Silica at Hypersonic Velocities," General Dynamics/Pomona, TM No. 6-346-337, 15 May 1963.

B. Felted Ceramics

Georgia Tech, in conjuntion with an industrial sponsor, previously carried out work on the development of felted ceramics. Orginially this program was directed towards the development of new types of ceramic building materials. As new compositions were investigated, it became evident that this type of material could have numerous applications, especially in the field of highly refractory materials.

The basic process makes use of the paper making techniques used in felting wall board and acoustical tile. Initial work was concerned only with the felting of slag wool, glass or refractory alumino-silicate fibers together with particulate material. After drying, the material is fired to the temperature required to develop the desired strength.

A primary advantage of this process is that it lends itself to the fabrication of large, relatively thin shapes of low and controllable density. Boards can be fabricated in continous length by 12 feet in width. Thickness may be varied nominally between 1/4-and 3/4-inch, with corresponding densities from 80 to 30 lb/ft^3.

The refractoriness of felted ceramic boards is normally limited by the high temperature properties of the fibers used. When very high temperature compositions are required for which fibers are not available, the process is modified by using the desired refractory in particulate form, felted with conventional wood fiber as a carrier. Such fibers are normally used in the fabrication of organic wall board and acoustical ceiling tile. After the refractory particulate material is felted with the wood fiber, the board is dried and then fired to the temperature required to sinter the

refractory material. During firing the wood fiber is burned out of the system. Aluminum oxide, zirconium oxide and magnesium oxide boards have been fabricated using this technique.

The background obtained at Georgia Tech and by the industrial sponsor in the development of felted ceramics indicated that essentially any ceramic composition which can be produced by conventional ceramic fabrication process, can be produced by this technique. However, only a limited amount of work was carried out on refractory applications.

The primary purpose of this phase of Project NOw 63-0143-d is to develop techniques and compositions of refractory felted ceramics for leading edge applications.

III. EXPERIMENTAL WORK

A. Slip-Cast Fused Silica Radomes

1. Flame Glazing

The system settings used in attempts to oxy-acetylene flame glaze cylinders, are given in Table I. These data are from trials during both the previous period and the period of this report.

The cylinders were cast in U. S. No. 1 Pottery plaster molds using conventional drain-cast techniques with no applied pressure. Except as noted in Table I, all cylinders were dried at 325°F with no subsequent pre-firing before glazing. After glazing the cylinders were extracted from the glazing furnace hot, with no annealing. Crazing of the glazed surface occurred in some cases.

2. Oxide Additions for Improved Strength and Thermal Properties

No work was conducted during this quarter with oxide additions to fused silica. The preliminary investigations under this Contract with oxide additions to fused silica suggested an improvement in the apparent refractoriness of the silica[3,4]. This work was continued under the Air Force Contract (Contract No. AF 33(657)-11504) to determine the relative merits of 11 different oxide additions, plus silicon carbide. The preliminary results of this work showed that under identical oxy-acetylene thermal environments, 11 of the additions improved the apparent ablation resistance of the base fused silica, while only one was detrimental. However, improvement was not large for the majority of the oxide additions.

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(3) Quarterly Report No. 1, Page 26.

(4) Quarterly Report No. 2, Page 19.

TABLE I

SLIP CAST FUSED SILICA OXYGEN-ACETYLENE
FLAME GLAZING DATA FOR TRIALS ON CYLINDERS

Cylinder Diameter	Flame O_2	Flame C_2H_2	Preheat Temperature	Stand-off Distance	Rotational Speed	Traverse Rate	Degree of Glaze *
(IN)	(CFH)	(CFH)	(°F)	(IN)	(RPM)	(IN/MIN)	
1	60	40	2050	1	23	5 3/4	1
1	60	40	2050	2	23	5 3/4	2
1	60	40	2050	3	23	5 3/4	3
1	60	40	2050	4	23	5 3/4	4
1	60	40	2050	5	23	5 3/4	6
1	60	40	2050	6	23	5 3/4	6
1	60	40	2050	4	9 1/4	2 7/8	1
1	60	40	2050	5	9 1/4	2 7/8	3
1	60	40	2050	6	9 1/4	2 7/8	5
2	120	80	2050	1	23	5 3/4	1
2	120	80	2050	2	23	5 3/4	3
2	120	80	2050	3	23	5 3/4	3

TABLE I (Cont.)

SLIP CAST FUSED SILICA OXYGEN-ACETYLENE
FLAME GLAZING DATA FOR TRIALS ON CYLINDERS

Cylinder Diameter	Flame O_2	Flame C_2H_2	Preheat Temperature	Stand-off Distance	Rotational Speed	Traverse Rate	Degree of Glaze*
(IN)	(CFH)	(CFH)	(°F)	(IN)	(RPM)	(IN/MIN)	
2	60	40	2050	1	23	5 3/4	4
2	60	40	2050	1 1/2	23	5 3/4	5
2	60	40	2050	2	23	5 3/4	5
2	90	40	2050	1	23	5 3/4	3
2	90	60	2050	1 1/2	23	5 3/4	3
2	90	60	2050	2	23	5 3/4	3
2	90	60	2050	2 1/2	23	5 3/4	4
4**	82	68	2050	1/2	25	4	8
4**	114	40	2050	1	25	4	7
4**	120	80	----	1/2	3	3/4	3
4**	120	80	----	1/2	5	1 1/4	5
4**	120	80	----	1/2	10	2 1/2	6

TABLE I (Cont.)

SLIP CAST FUSED SILICA OXYGEN-ACETYLENE
FLAME GLAZING DATA FOR TRIALS ON CYLINDERS

Cylinder Diameter	Flame O_2	Flame C_2H_2	Preheat Temperature	Stand-off Distance	Rotational Speed	Traverse Rate	Degree of Glaze*
(IN)	(CFH)	(CFH)	(°F)	(IN)	(RPM)	(IN/MIN)	
7**	114	40	2050	1	25	4	8
7	120	80	2050	1/2	4 1/4	1 1/8	1
7	120	80	2050	1	4 1/4	1 1/8	2
7	120	80	2050	1 1/2	4 1/4	1 1/8	3
7	120	80	2050	1 1/2	9 1/2	3 7/8	6
7**	120	80	2050	1 1/2	7 1/4	2 1/2	5
7**	120	80	2050	1 1/2	9 1/4	2 7/8	6
7**	120	80	2050	2	9 1/4	2 7/8	6

*Code: 1. Extreme Boiling; 2. Heavy Boiling; 3. Boiled; 4. Light Boiling; 5. Excellent Glaze; 6. Fair Glaze; 7. Light Sheen; 8. No Glazing

**Pre-fired at 2200°F for 3 1/2 hours.

Only additions of CrO_2 and TiO_2 improved ablation resistance sufficiently to be considered for further study. Closer evaluation of the TiO_2 addition showed the effect to be pseudo in nature and to be caused by a frothing of the composite material.

B. Felted Ceramic Compacts

1. Felting Techniques

The major work of this quarter was directed toward the development of techniques and compositions for preparing refractory felted ceramic compacts.

Since the state of the art of refractory fiber development has not reached the point where refractory inorganic fibers have sufficient high temperature properties above 3000°F, it was decided that first efforts would be made using only wood fiber and refractory particulate material. Two methods developed under the previous contract sponsored by the industrial firm seem to hold promise. In the first method, the wood fiber acts only as a binder in the green (unfired) state and is completely lost on firing. In the second method, the porous wood fiber is pre-treated with a colloidal refractory oxide suspension or with a soluble compound which gives a refractory material upon heating. The treated fibers are then felted with refractory particulate material in the normal manner, dried and fired. The fibers burn out on firing but are replicated by the materials with which they were pre-treated.

A 4-inch diameter compact 1/4 to 3/4 inch in thickness was taken as the optimum size for laboratory felting and firing of samples. Equipment for felting and pulling a vacuum on the compacts to aid in their draining is shown in Figures 1, 2 and 3. The cylinder to hold the water suspension of fiber and particulate

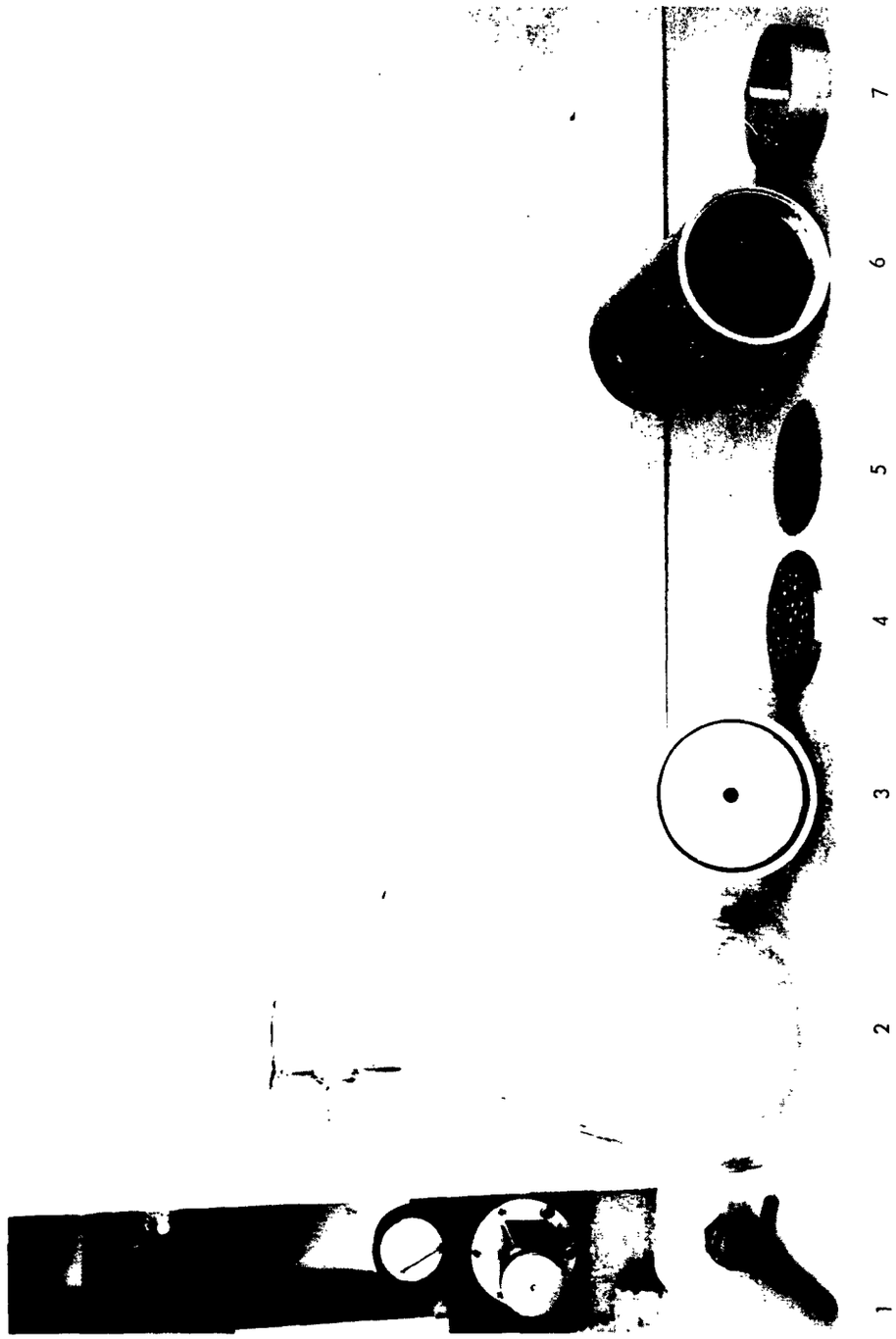

Figure 1. Disassembled Melting Apparatus.

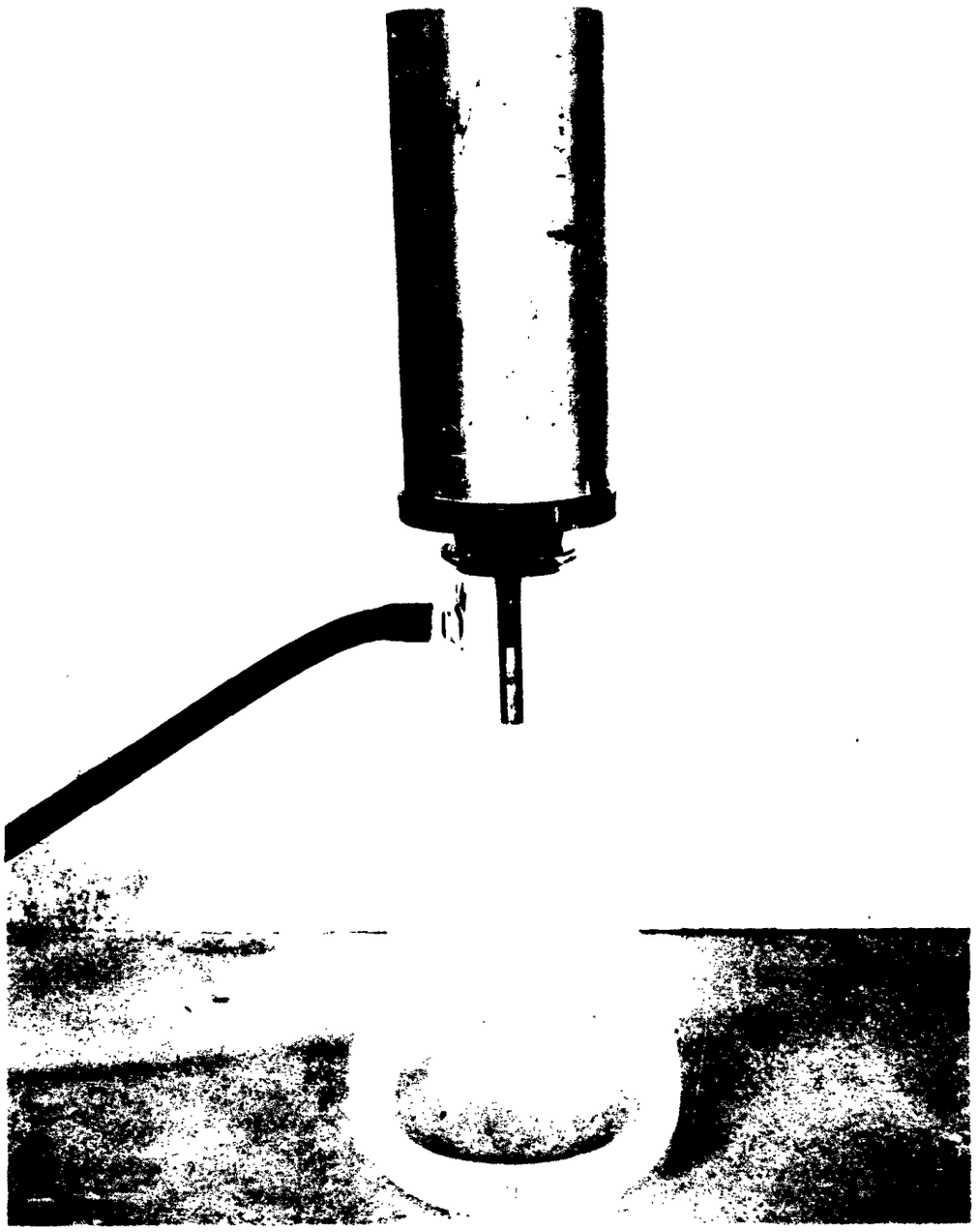

Figure 2. Folin Apparatus Ready for Use.

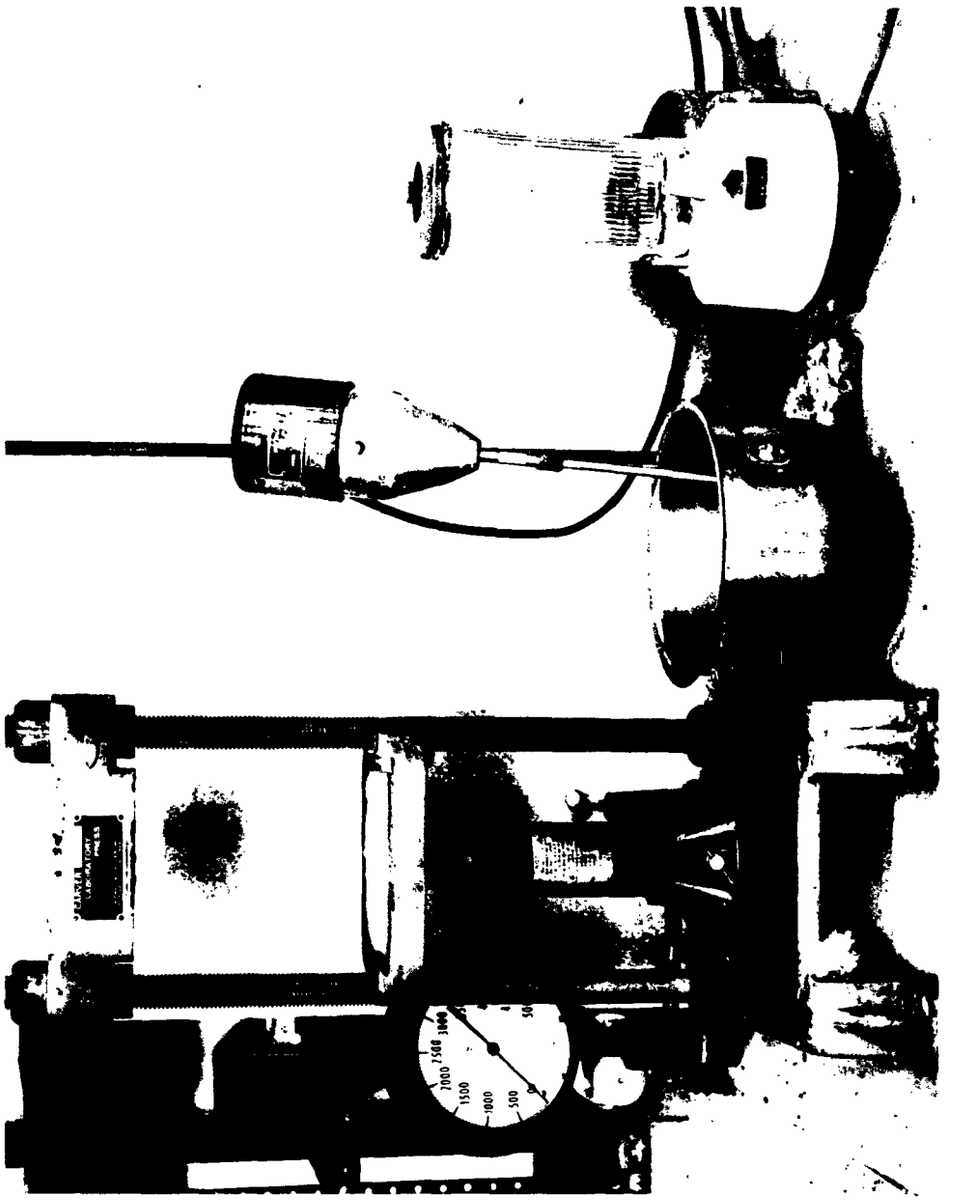

Figure 3. Related Accessory Equipment for Forming Felted Compacts.

material has an inside diameter of 4-inches and is screwed to the base to form a vacuum tight seal. The disassembled cylinder showing all components is seen in Figure 1. The parts are assembled by screwing the cylinder (6) to the base plate (3). The perforated aluminum plate (4) rests inside the cylinder on a shoulder of the base plate. A 40 mesh stainless steel screen (5) is placed over the perforated aluminum plate. The assembled cup is attached to the filter flask (2) through a rubber stopper, and then the flask is attached to the aspirator (1). After felting, the base plate is removed and the perforated plate, screen, and felted compact are removed as a unit with the plunger (7). The assembled apparatus is shown in Figure 2. Figure 3 shows accessory equipment needed for forming the slurry and pressing the compacts to a higher density.

A few compacts were made from previously successful compositions formulated by the industrial sponsor. These were made in order to gain competence and experience in felting operations since only the firing and testing operations of such felts were carried out at Georgia Tech.

The first compact made was one involving pre-treatment of the wood fiber with a soluble Zirconium salt. The procedure followed is outlined below.

Twenty grams of $ZrOCl_2$ was dissolved in 100 ml of Methanol and 10 grams of wood fiber was soaked in the resulting solution. One hundred grams of -200 mesh ZrO_2 was suspended in one liter of water with the aid of an electric stirrer. The $ZrOCl_2$ soaked wood fiber was then added to the suspension along with 20 ml of concentrated NH_4OH and thoroughly dispersed by stirring vigorously for five minutes. The mixture was then poured into a section of aluminum pipe placed inside a Gooch crucible and a vacuum pulled on the system. Draining was rapid with the majority of the particulate material passing through the

screen before the fibers had a chance to form a mat in the bottom of the cylinder. The compact that was formed was removed and pressed on a hydraulic press to a pressure of 16 psi. This produced a compact after drying over night with a density of 49 lb/ft^3 indicating only a small amount of the ZrO_2 was retained on the fibers. Upon firing this compact fell apart due to the small amount of particulate material bridging between fibers.

Eight new compositions were formulated as shown in Table II. The same procedure was followed for all compacts as outlined above except that there was no pre-treatment of fibers. The amount of water in these compacts was cut to 500 ml so that the slurries had initial solid contents of 30 to 33 per cent. This water reduction produced a pasty consistency and the compositions had to be raked into the Gooch crucible for forming the compacts. Also, the advantage of the fiber water laying action was lost. The drainage rate on these compacts was very rapid and the compacts produced were somewhat more dense than the initial compact due to the fact that far less particulate material escaped through the wire screen. The weight and bulk density of the dried compacts appear in Table III.

The inhomogeneity due to poor felting action was very apparent after firing the compacts at 2300°F overnight (15 hours). The compacts were very fragile and dusty.

The rapid drain times of all previous experiments and examination of the dried compacts indicated that the action of the laboratory electric stirrer was not sufficient in breaking down bundles and clots into individual wood fibers. However, individual fibers were later obtained by using a Waring Blendor. The blendor also tends to fibrillate the individual fibers.

TABLE II

COMPOSITION OF FELTED COMPACTS

Compact No.	1	2	3	4	5	6	7	8
Wood fiber	20	40	20	40	20	40	20	40
Ball clay	30	30	50	50	100	100	74	74
Zirconia	180	180	160	160				
Alumina					100	100	148	148

NOTE: All compacts were made with 500 ml of water.
Tabulated quantities are in grams.

TABLE III

WEIGHT AND BULK DENSITY OF DRIED COMPACT FROM TABLE I

Compact No.	Diameter (IN)	Avg. Thickness (IN)	Weight (GM)	Bulk Density (LB/FT^3)
1	4.0	0.39	59.2	44.8
2	4.0	0.80	159.1	60.3
3	4.0	0.43	80.0	58.1
4	4.0	0.66	102.2	47.9
5	4.0	0.53	113.0	65.8
6	4.0	0.81	188.9	55.9
7	4.0	0.65	154.6	72.3
8	4.0	0.80	177.1	67.2

An additional composition was formulated, Compact No. 9, and a compact was made using the procedure outlined below:

Compact No. 9: 10 gm Wood fiber, 2 gm Starch, 60 gm Alumina
1 gm Alum, 1 ml Ammonium hydroxide, 1000 ml Water

Five grams of the wood fiber and 40 ml of water were mixed in a Waring Blendor for 15 seconds then trensferred to a 4-liter beaker. Mixing was continued with an electric stirrer. The remaining 5 grams of wood fiber and additional 400 ml of water were mixed in the Waring Blendor 15 seconds, then transferred to the 4 liter beaker. The remaining 200 ml of water and other materials were added and thoroughly mixed, then poured into the aluminum cylinder in the Gooch crucible. Drain time was greater than 10 minutes for t. .s compact. Dry density of this compact was 47.7 lb/ft^3.

The same procedure as above was repeated with Composition 1 of Table I. This compact had a drain time greater than one hour even though a vacuum was pulled on it. A strong dry compact was produced with a density of 64.5 lb/ft^3.

After completion of the above preliminary experiments, it was thought that sufficient competence and experience had been gained to begin pursuit of more intensive felting studies. A composition which had previously been used and which produced compacts with good strengths was used for these new studies. The effect on drain time, green and fired strength, and density by such variables as fibrillation, slurry concentration, flocculants, ball clays, wetting agents and vacuum drain time were to be evaluated.

The composition selected for the above studies has the following ratios of materials: 1 part wood fiber; 2 parts -325 mesh alumina; 2 parts Ball clay. A comparison of drain time and green density with fibrilation and type of ball clay used was made by preparing eight compacts with the above formulation. All compacts were made with 15 grams of wood fiber and 1500 ml. of water giving a solids content in the slurry of 4.7 per cent. Four of the compacts were made with each clay after respectively blunging the fiber in the Waring Blendor for five, ten and fifteen seconds. Data obtained on these compacts are shown in Table IV.

TABLE IV

EFFECT OF FIBER REFINING AND TYPE OF BALL CLAY ON DRAIN TIME AND GREEN DENSITY OF COMPACTS FELTED IN PORCELAIN GOOCH CRUCIBLE

Compact	Ball Clay	Blunging Time (SEC)	Drain Time (SEC)	Dry Weight (GM)	Average Thickness (IN)	Dry Density (LB/FT^3)
I A	Stratton	0	40	26.0	.47	16.7
II A	Stratton	5	60	32.7	.47	21.1
III A	Stratton	10	85	38.6	.47	24.9
IV A	Stratton	15	590	35.6	.48	22.5
I B	C and C	0	520	42.4	.49	26.2
II B	C and C	5	945	41.3	.42	29.8
III B	C and C	10	1200	37.5	.39	29.1
IV B	C and C	15	INFINITE	-	-	-

The compacts in Table IV and all previous compacts were felted in a 4.0 inch I.D. aluminum cup placed inside a porcelain Gooch crucible which in turn was fastened to a filter flask on which a vacuum was pulled by a water aspirator. Much of the effect of the vacuum was lost by air being pulled in between the walls of the aluminum cylinder and the porcelain crucible. At this point the apparatus shown in Figures 1 and 2 was constructed to obtain a better vacuum on the slurry being felted. The compacts shown in Table IV were remade using the new apparatus. The compacts made without blunging of the fiber in the Waring Blendor were eliminated because of their short drain time. Data for these compacts are in Table V.

TABLE V

EFFECT OF FIBER REFINING AND TYPE OF BALL CLAY

ON DRAIN TIME AND GREEN DENSITY OF COMPACTS FELTED IN ALUMINUM CUP

Compact No.	Ball Clay	Blunging Time (SEC)	Drain Time (SEC)	Dry Weight (GM)	Average Thickness (IN)	Dry Density (LB/FT^3)
II C	Stratton	5	62	33.1	0.42	23.9
II C	Stratton	10	230	41.5	0.48	26.2
IV C	Stratton	15	200	40.7	0.34	36.3
II D	C and C	5	145	36.8	0.38	29.4
III D	C and C	10	270	40.0	0.42	28.9
IV D	C and C	15	560	36.2	0.33	32.6

As can be seen from Tables IV and V the drain time for the C and C ball clay compacts was greatly decreased by the better vacuum obtainable with the new felting apparatus.

In an attempt to increase the density of compacts, two slurries as previously made after blunging the wood fiber 10 and 15 seconds in the Waring Blendor were treated with 37 grams of a solution made by dissolving 15 grams of aluminum sulfate in 250 ml of water. The slurries were further treated with 15 grams of a solution made by dissolving 50 grams of Trilinoleic acid and 12.75 grams of Potassium hydroxide in 247.75 grams of water.

The flocculating action of these two solutions greatly increased the drain times of the compacts but also caused the fibers to retain much of the particulate material with a resulting increase in dried density of the felts. The density of the two felts prepared were 40.9 and 48.6 lb/ft^3 respectively for the compacts prepared by blunging the wood fiber in the Waring Blendor 10 and 15 seconds.

In order to improve felting characteristics and retention of particulate material a more dilute slurry was prepared. The experiment outlined previously for which data is reported in Table V was repeated using C and C Ball Clay and a slurry solids concentration of 2 per cent. Drain time was increased for the larger amount of liquid used, however, there was no apparent change in green density.

A 1: 2: 2 composition of wood fiber; clay; alumina was prepared using 10 grams of wood fiber and 20 grams each of clay and alumina in 2000 ml of water. The wood fiber was not blunged in the Waring Blendor. One ml of concentrated ammonium hydroxide and one ml of a 0.05 per cent solution of a

polyacrylamid flocculating agent were added to 500 ml of water and thoroughly mixed then slowly stirred into the fiber-clay-alumina-water slurry, giving a final solid contents of 2 per cent in the slurry. Felting of this compact gave a drain time of 93 seconds but a green density of only 22.8 lb/ft^3.

The above experiment was repeated exactly except the ammonia and the polyacrylamid flocculating agent was diluted with 250 mls of water and one gram of alum was dissolved in the other 250 mls of water. These two solutions were added to the slurry with slow stirring until they were thoroughly mixed into the slurry. The alum solution was added first. Upon pouring, the drain time was reduced to 60 seconds with a thick filter cake being built up. Data on the dried density was not obtained in time for inclusion in this report.

2. Firing of Felts

All firing of the felted compacts has been in a globar furnace. The compacts are fired to 2500°F and held for 4 hours at temperature, then allowed to cool in the furnace to room temperature. Because of the wood fiber burning out, the lower portion of the firing schedule is critical. Too rapid firing will result in disruption of the compact due to gases being vaporized from the wood fiber. The firing schedule used for firing the first 4-inch compacts is shown in Table VI. The times shown in this table are the times at which the furnace controller is increased and is not the actual rate of rise over the period indicated.

TABLE VI

SCHEDULE FOR FIRING FELTED COMPACTS

Temperature (°F)	Time (MIN)
250°F	0
300°F	15
340°F	25
360°F	35
380°F	45
400°F	55
440°F	65
480°F	75
520°F	85
550°F	95
2500°F	125

*Note furnace is turned up after 30 minutes at 550°F or after all smoking has ceased.

The reason for requiring an hour and 35 minutes to reach temperature is due to the design of the furnace. The globars are exposed inside the furnace and if they become too hot, radiation will ignite the wood fiber in the felts. A quicker method of firing has been adopted with no apparent damage to the compacts. The furnace is preheated and allowed to stabilize at 550°F. The

furnace is shut off and the dried compacts placed in it. They are then allowed to remain 45 minutes or until all smoking has ceased, whichever is longer. The furnace is then increased to 2500°F at a rapid rate. The furnace requires approximately one hour to reach 2500°F from an initial temperature of 550°F.

All of the initial compacts were very fragile and fell apart on handling or under their own weight. This was due to their low densities.

The re-run of the compact made from Composition 1 of Table I by blunging the fiber in a Waring Blendor and using a slurry containing 1000 ml of water produced, upon firing at 2500°F for 4 hours, a strong but dusty body. It had a density of 74.7 lb/ft^3 and a linear shrinkage of 16.3 per cent. Due to Zirconia's high sintering temperature, it will be necessary to refire this compact above 3000°F to develop maximum strength.

Of the compacts fired thus far, as a general rule, it may be stated that only those compacts with a green density greater than 40 lb/ft^3 produce satisfactory fired bodies. The compacts listed in Table III are an exception to this rule because they were not true felts. These compacts were formed from a pasty slurry rather than a dilute solution.

The two compacts made by blunging the fibers in a Waring Blendor for 10 and 15 seconds, then flocculating with alum and trilinoleic acid solutions, 2 per cent solids concentration slurries, and with felted green densities of 40.9 and 48.6 lb/ft^3 respectively gave the best fired bodies yet obtained. These bodies appear to have excellent strength and fired densities of 50.7 and 58.8 lb/ft^3 respectively. However, shrinkage was 11.7 and 12.5 per cent. This is much higher than desired and will probably decrease as the clay content decreases. Figure 4 shows two unfired and two fired compacts. The firing shrinkage

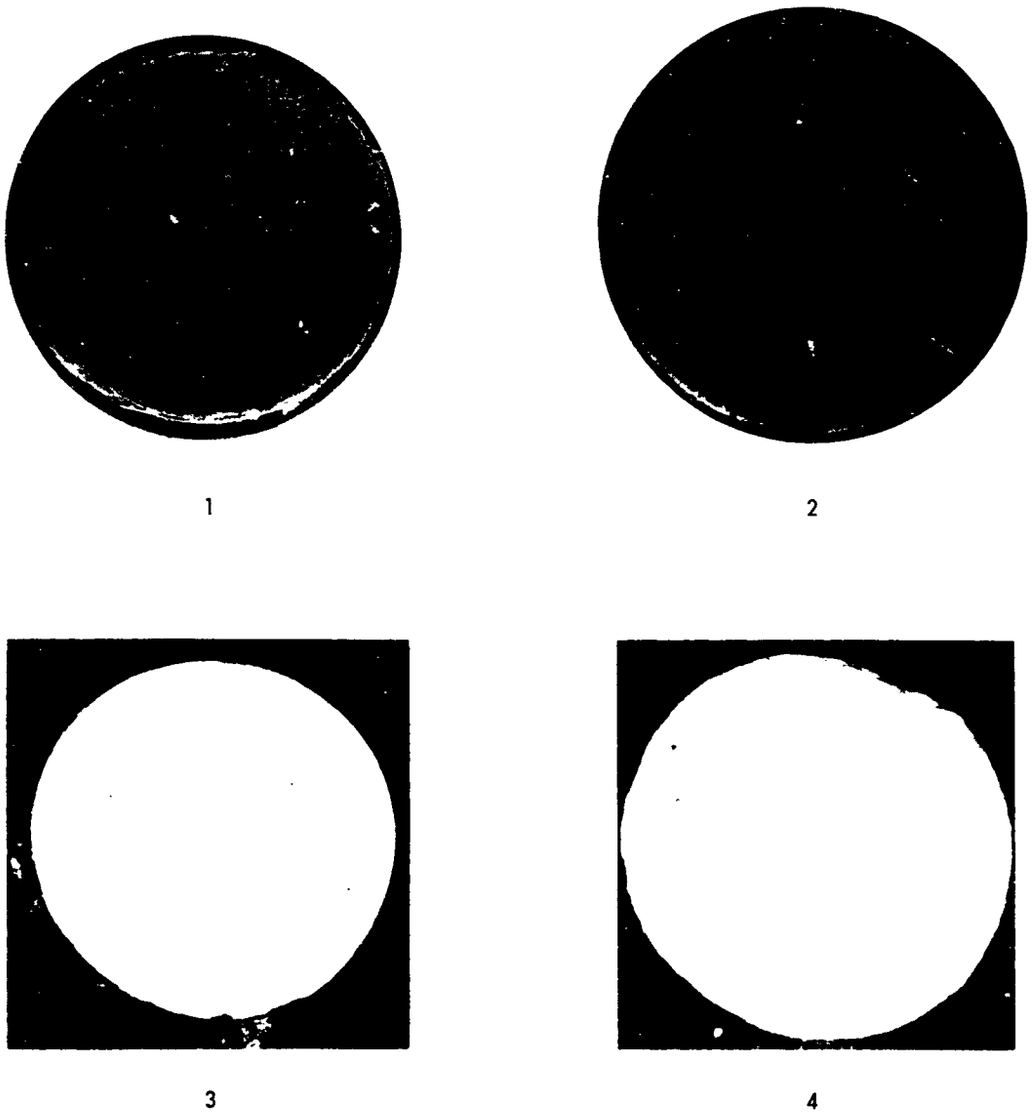

Figure 4. Dried and Fired Felted Compacts.

can be readily noted. Physical data on the strength of these compacts was not available in time for inclusion in the report. Specimens flocculated with the polyacrylamid agent alone and with the agent and alum have not yet been fired.

IV. DISCUSSION OF RESULTS

A. Slip-Cast Fused Silica Radomes

1. Flame Glazing

As expected from the relative heat flux curves of the oxy-acetylene flame, this flame affords a high degree of parameter flexibility not present with the arc-plasma flame[5]. The versatility of the flame for glazing slip-cast fused silica is illustrated in Table I. This data, while not being complete in nature, suggested that complete flame glazing of a large radome shape, approximately 31 inches tall with a base diameter of approximately 14 inches, could be accomplished with a rotational speed of 5 RPM or less; the variables being stand-off distance and fuel gas flow rates. This was attempted under Air Force Contract No. AF 33(657)-11504 and was found to be the case. In this, however, only the stand-off distance was varied with constant fuel flow and rotation. Complete glazing of the large shape was accomplished with 5 RPM rotation and with a flame of 105 CFH O_2 and 70 CFH C_2H_2.

B. Felted Ceramic Compacts

Upon selection of the basic composition of 1 part wood fiber: 2 parts clay: and 2 parts alumina, it was not expected that this composition would give very good felts without the aid of some flocculating agent. However, it was felt that the results of felting this composition alone was needed as a standard in order to evaluate the variables of dilution of slurry,

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type of ball clay used and effect of fibrillation or refining of the fibers on the retention of particulate material on and around the fibers. Only after obtaining this basic data could the effect of various flocculating agents be effectively evaluated.

Data at the time of this report are not complete enough to be conclusive; however, from the data thus far collected, it is thought that the separating and fibrillating of the fibers in the Waring Blendor aids in the retention of particulate material. A strong dense body was developed in this manner with Composition 1 of Table I. However, the drain time was excessively long and would prohibit this composition in being used in conventional felting machine operations. In all cases blunging of the fiber in the Waring Blendor increased drain time. At present, some separation and fibrillation of the fibers appears necessary, however, further studies with more dilute slurries may eliminate the need for this step.

Dilution of the slurries to 2 per cent solids increased particulate material retention with and without flocculating agents. At this time it is believed that a 2 per cent slurry is the highest concentration practical. During the next interval slurry concentrations down to 1/2 per cent will be investigated as a means of increasing particulate material retention.

Data on the effect of clay type is not complete but experimental evidence indicates that the Stratton clay gives a much faster drain time than does the C and C clay. The type of clay does not apparently affect the green density. As new compositions are formulated containing smaller percentages of clay, two problems should be lessened. Both the drain times and firing shrinkage will be decreased.

Of the compacts felted with a flocculating system only those using the combination of alum and trilinoileic acid have been fired. These gave excellent compacts, however, linear shrinkage was somewhat greater than desired. At this time the effect of various type of flocculating agents on green density and drain time cannot be evaluated, however, the data is indicative that a flocculating system will be necessary.

V. PROGRAM FOR THE NEXT INTERVAL

The studies of the effects of the following variables, fiber separation and fibrillation, type of clay and deflocculating system on a 2 per cent slurry of a 1: 2: 2 composition of fiber; clay; alumina will be completed.

The best techniques and material systems developed will be used. New fiber-clay-alumina compositions will be investigated with decreasing amounts of clay or other binder materials in attempts to approach a 100 per cent alumina-wood fiber composition with good green and fired properties. If fired strengths can be developed in high alumina bodies organic binders will be considered where necessary to improve green handling qualities.

Wood fibers will be pre-treated with aluminum chlorhydroxide solutions and suspensions of colloidal alumina (Baymal) to increase the densities of alumina-fiber systems.

A parallel study on zirconia-wood fiber systems will be conducted with felted specimens fired to temperatures of 3500°F. Also MgO and Cr_2O_3 will be considered separately and as spinel systems.

VI. PERSONNEL

The personnel assigned to the project and the approximate time devoted to the work of the project by each are listed below:

N. E. Poulos	Project Director	1/4 time
J. D. Walton	Special Research Engineer	1/10 time
C. A. Murphy	Assistant Project Director	1/4 time
J. N. Harris	Assistant Research Engineer	1/3 time
E. A. Welsh	Student Assistant	1/3 time

Respectfully submitted,

N. E. Poulos
Project Director

Approved:

J. D. Walton, Head
High Temperature Materials Branch

F. Bellinger.
F. Bellinger, Chief
Chemical Sciences and Materials Division

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